PATENT ABSTRACTS OF JAPAN

(11)Publication number:

09-007810

(43) Date of publication of application: 10.01.1997

(51)Int.CI.

H01F 1/053 H01F 41/02

(21)Application number : 07-151238

(71)Applicant: SHIN ETSU CHEM CO LTD

(22)Date of filing:

19.06.1995

(72)Inventor: TAKAGUCHI KAZUHIRO

YOSHIKAWA MASAO

(54) HIGH CORROSION RESISTANT PERMANENT MAGNET AND ITS MANUFACTURING METHOD (57) Abstract:

PURPOSE: To improve the corrosion resistance by a method wherein low hardness metal plating layer and high hardness metal plating layer are formed on the surface of an R-Fe-B base permanent magnet. CONSTITUTION: Within the title high corrosion resistant permanent magnet, low hardness metal plating layer and high hardenes metal plating layer are formed on the surface of an R-Fe-B base permanent magnet (R represents at least one kind of rare earth elements including Y). Besides, the low hardness metal plating layer is a non-gloss plating layer and the high hardness metal plating layer is gloss nickel plating layer while the film thickness ratio between the low hardness metal plating layer and the high hardness metal plating layer is 6:4-8:2. Furthermore, in order to manufacture this permanent magnet, after coating the surface of R-Fe-B base permanent magnet with low hardness metal plating layer, mechanical impulse is given to the permanent magnet to stop up the pinholes in the low hardness metal plating layers to be coated with high hardness metal plating layer later.

LEGAL STATUS

[Date of request for examination]

25.02.1998

[Date of sending the examiner's decision of rejection] 04.11.1999

[Kind of final disposal of application other than the

examiner's decision of rejection or application

converted registration]

[Date of final disposal for application]

[Patent number]

3234448

[Date of registration]

21.09.2001

[Number of appeal against examiner's decision of

11-19350

rejection]

Searching PAJ Page 2 of 2

[Date of requesting appeal against examiner's 06.12.1999 decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

* NOTICES *

Japan Patent Office is not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to a rare earth permanent magnet especially the R-Fe-B system permanent magnet which has high corrosion resistance, and its manufacture method. [0002]

[Description of the Prior Art] The rare earth permanent magnet is used abundantly by outstanding magnetic properties in the field of the electrical and electric equipment, and the high performance-ization is demanded increasingly in recent years. The inside of this and a R-Fe-B system permanent magnet are conventional R-Co. Nd which is the main element of a R-Fe-B system permanent magnet as compared with a system permanent magnet R-Co Sm which is the main element of a system permanent magnet Co by which existing in abundance in resource and supply are not stabilized Since it is not used in large quantities, a material cost is cheap, and magnetic properties are also R-Co. Since it is the extremely excellent permanent magnet material which easily endures a system permanent magnet Former R-Co It is not only substituted for the small magnetic circuit where the system permanent magnet has been used by this, but it is going to be widely applied to the field for which the hard ferrite or the electromagnet was used from the cost side. However, this R-Fe-B system permanent magnet has the defect of reacting very easily with the humidity in atmospheric air, and oxidizing. Although corrosion advances through the grain boundary inside from the surface and an oxide not only generates oxidation, but it produces the so-called phenomenon of grain boundary corrosion on the magnet surface, this is because very activity R rich phase exists in the grain boundary of a R-Fe-B system permanent magnet. If deterioration of very big magnetic properties is caused and corrosion advances at the time of practical use, the corrosion of a grain boundary will reduce the engine performance of the device incorporating a magnet, and the problem of polluting the device circumference will produce it.

[0003] Although various kinds of surface treatment methods are proposed in order to conquer the defect of such a R-Fe-B system permanent magnet, especially nickel plating is resin paint and aluminum. Since it has the advantage with very little hygroscopicity of the enveloping layer itself with a high mechanical strength that there are few pinholes, compared with ion plating, chemical conversion, and other surface treatment, generally it is used widely. [0004] However, although there are few pinholes, it does not go to the reason for saying that there is no pinhole, but various composite platings are proposed aiming at the further corrosion-resistant improvement. For example, in JP,1-268004,A, mat metal plating is performed as a substrate and improvement of corrosion-resistant [preparing an enveloping layer with few pinholes on it] is in drawing. Moreover, at JP,1-42805,A, it is Cu. Corrosion resistance is improved by preparing a layer and the double layer of nickel-P. Furthermore, there are some which aimed at high corrosion resistance by carrying out the laminating of the layer to which the sulfur concentration in a nickel-plating layer was changed like JP,2-23603,A and JP,4-253306,A. In addition, the composite plating of various combination is proposed. after [furthermore,] performing nickel plating -- a corrosion-resistant resin layer and corrosion resistance -- formation -- the thing with a coat is also proposed.

[0005] [Problem(s) to be

[Problem(s) to be Solved by the Invention] However, the demand to the corrosion resistance of a R-Fe-B system permanent magnet becomes severer recent years still, and it is necessary to improve corrosion resistance further rather than the above-mentioned technique. [0006]

[Means for Solving the Problem] this invention persons resulted in this invention, as a result of striving wholeheartedly to solve said technical problem. That is, this invention consists of following the (1) - (7), and solves said trouble.

(1) A high corrosion resistance permanent magnet characterized by carrying out the laminating of the metal plating layer with a high degree of hardness to a metal plating layer with a low degree of hardness on it on the surface of a R-

Fe-B system permanent magnet (rare earth elements containing Y are kinds at least for R).

- (2) A high corrosion resistance permanent magnet given in the above (1) characterized by for a metal plating layer with a low degree of hardness being a mat nickel-plating layer, and a metal plating layer with a high degree of hardness being a gloss nickel-plating layer.
- (3) The above (1) whose ratio of thickness of a metal plating layer and thickness of a metal plating layer with a high degree of hardness with a low degree of hardness is characterized by being 6:4-8:2, or a high corrosion resistance permanent magnet given in (2).
- (4) A manufacture method of a high corrosion resistance permanent magnet characterized by giving a mechanical impact to this permanent magnet, crushing a pinhole of a metal plating layer with a low degree of hardness, and subsequently to a it top covering a metal plating layer with a high degree of hardness after covering a metal plating layer with a low degree of hardness on the surface of a R-Fe-B system permanent magnet (rare earth elements containing Y are kinds at least for R).
- (5) A manufacture method of a high corrosion resistance permanent magnet characterized by covering a metal plating layer with a low degree of hardness on the surface of a R-Fe-B system permanent magnet (rare earth elements containing Y are kinds at least for R), crushing a pinhole of a metal plating layer with a low degree of hardness on it, giving a mechanical impact to this permanent magnet, and subsequently to a it top covering a metal layer with a high degree of hardness on it.
- (6) The above (4) characterized by for a metal plating layer with a low degree of hardness being a mat nickel-plating layer, and a metal plating layer with a high degree of hardness being a gloss nickel-plating layer, or a manufacture method of a high corrosion resistance permanent magnet given in (5).
- (7) a degree of hardness -- being low -- a metal -- plating -- a layer -- thickness -- a degree of hardness -- being high -- a metal -- plating -- a layer -- thickness -- a ratio -- 6:4 8:2 -- it is -- things -- the feature -- ** -- carrying out -- the above -- (-- four --) (-- six --) -- either -- a publication -- high -- corrosion resistance -- a permanent magnet -- manufacture -- a method.

Below, this invention is explained in detail.

[0007] A high corrosion resistance permanent magnet of this invention has a double layer which becomes the surface from a metal plating layer with a low degree of hardness, and a metal plating layer with a high degree of hardness. Although measured with a Vickers hardness plan etc., a degree of hardness of a metal plating coat must measure it, as effect of a base is avoided, when measuring a degree of hardness of a metal plating coat with a Vickers hardness plan. A metal plating layer with a low degree of hardness of this invention is Vickers hardness (Hv) 500. A metal plating layer with a high degree of hardness has good Vickers hardness (Hv) 500-1500 hereafter. Vickers hardness (Hv) of a metal plating layer with a low degree of hardness is 500. Since a metal plating coat does not cause plastic deformation, a pinhole does not collapse upwards and it becomes easy to generate a crack etc. on a metal plating coat by mechanical shock when it exceeds, and a mechanical shock is added, it is unsuitable. Moreover, Vickers hardness (Hv) of a metal plating layer with a high degree of hardness is 500. Since it becomes easy to produce a crack in plating when 1500 is exceeded, since abrasion resistance on the surface of a permanent magnet cannot be secured and it becomes easy to attach a crack, when it is the following, it is not desirable.

[0008] As a metal plating layer with a low degree of hardness in this invention, there are a mat nickel-plating layer, a copper-plating layer, a galvanization layer, etc., and there are a gloss nickel-plating layer, a nickel alloy plating layer, a non-electrolyzed nickel-Lynn plating layer, a chrome plating layer, etc. as a metal plating layer with a high degree of hardness. Although a double layer is formed in the permanent magnet surface in various combination out of these, combination of a mat nickel-plating layer and a gloss nickel-plating layer is desirable.

[0009] Although a double layer of a metal plating layer with a low degree of hardness and a metal plating layer with a high degree of hardness is prepared in the R-Fe-B system permanent magnet surface in this invention In case the coat is prepared, a pinhole which prepares a metal plating layer with a low degree of hardness in the R-Fe-B system permanent magnet surface, subsequently adds a mechanical impact first, and exists in a metal plating layer with the low degree of hardness is crushed, the permanent magnet surface and atmospheric air are intercepted, and a metal coat with a high degree of hardness is covered after that. Or giving a mechanical impact, a metal plating layer with a low degree of hardness is covered on the permanent magnet surface, a pinhole is crushed and, subsequently a metal coat with a high degree of hardness is covered.

[0010] In order to give a mechanical impact to a metal plating layer with a low degree of hardness, there are methods, such as throwing a shot with a diameter of about 1-30mm at a metal plating layer with a low degree of hardness. However, cautions are required in order to damage a permanent magnet object, if mechanical shock force is too large. Since impulse force which a permanent magnet object damages changes greatly with configurations, sizes, etc. of the permanent magnet object, it is difficult to carry out numerical limitation. Therefore, when giving mechanical shock

force to a permanent magnet object, it is desirable to check in advance that a permanent magnet object which is a processed material is not damaged according to the impulse force. Moreover, when mechanical shock force is too weak, a pinhole does not collapse and an effect of this invention is not acquired.

[0011] As for a ratio of thickness of a metal plating layer, and thickness of a metal plating layer with a high degree of hardness with a low degree of hardness of the R-Fe-B system permanent magnet surface by this invention, being referred to as 6:4-8:2 is desirable. It is for corrosion resistance to improve further within the limits of this. Moreover, a metal plating layer with a low degree of hardness is [1-20 micrometers and 1-15 micrometers of desirable 5-15 micrometers and desirable metal plating layers with a high degree of hardness of thickness of a corrosion-resistant metal coat of a duplex] 2-10 micrometers preferably, respectively, and 5-30 micrometers is suitable for thickness of the sum total of a metal plating layer of these duplexes. If sum total thickness is thicker than 30 micrometers, time amount and the amount of drugs which plating takes are great, and since costs start too much, it is not practical. In less than 5 micrometers, a plating film will be too thin and corrosion resistance will deteriorate.

[0012] A R-Fe-B system permanent magnet of this invention uses R (for R, rare earth elements containing Y are kinds at least), Fe, and B as main elements, and, as for the presentation, it is desirable that 5 - 40 % of the weight and Fe are [50 - 90 % of the weight and B] 0.2 - 8 % of the weight for R. An amount of R is alpha-Fe at less than 5 % of the weight. If the amount of deposits increases too much, and high coercive force is not acquired and it exceeds 40 % of the weight, a nonmagnetic phase containing R will increase too much, and a residual magnetic flux density will fall. Fe An amount is alpha-Fe, when [than 90 % of the weight] more [a residual magnetic flux density is low at less than 50 % of the weight, and a magnet property is not acquired and]. The amount of deposits increases too much and high coercive force is not acquired. if coercive force is not acquired at less than 0.2 % of the weight but there are more amounts of B than 8 % of the weight -- B -- a rich nonmagnetic phase increases too much and a residual magnetic flux density falls. Moreover, what added elements, such as C, aluminum, Si, Ti, V, Cr, Mn, Co, nickel, Cu, Zn, Ga, Zr, Nb, Mo, Ag, Sn, Hf, Ta, and W, for a magnetic-properties improvement is contained in this invention. Co of an addition of these alloying elements is 0.5 - 20 % of the weight preferably 30 or less % of the weight, and other alloying elements are good to consider as 8 or less % of the weight in total. Co Although it adds for an improvement of a residual magnetic flux density, if the amount exceeds 30 % of the weight, coercive force will decline. Since magnetic properties will deteriorate if it exceeds 8 % of the weight in total, other alloying elements should be avoided.

[0013] After blending a raw material so that it may become the above presentations, using a RF fusion furnace etc., dissolving and casting, producing an ingot and carrying out coarse grinding of the ingot with a jaw crasher, a stamp mill, etc., it pulverizes with a ball mill, a jet mill, etc., and impalpable powder with a mean particle diameter of 1-20 micrometers is obtained. It fabricates all over a magnetic field, and this impalpable powder is sintered at 1000-1250 degrees C for 0.5 to 10 hours, and, finally is heat-treated by 400 - 900 **, and a R-Fe-B system permanent magnet is manufactured. In addition, in order that a R-Fe-B system alloy may tend [very] to oxidize, the above-mentioned production process is performed in an inert atmosphere in a vacuum or argon gas etc.

[Function] According to this invention, a metal plating layer with a degree of hardness low as the 1st layer is given to the R-Fe-B system permanent magnet surface, in order to add a mechanical impact and to crush the pinhole which exists in the layer [1st] metal plating layer, the pinhole of the 1st layer is closed and the permanent magnet surface is completely intercepted with atmospheric air. However, since just a metal plating layer with a low degree of hardness is not enough as a mechanical strength, the abrasion resistance of the surface lining of a permanent magnet is further secured by giving a metal coat with a high degree of hardness on it.

[Example] Although an example is given and the embodiment of this invention is explained concretely hereafter, this invention is not limited to these.

It is 32Nd-59.3Fe-7Co-1.2 B-0.5 aluminum in an example 1 - 5 % of the weight of examples. The induction-heating RF fusion furnace was used, in the argon ambient atmosphere, it dissolved, each raw material metal beyond purity 99.9wt% was cast, and the alloy ingot was produced so that it might be formed. After performing homogenization heat treatment of 1100 degree-Cx 24 hours in an argon ambient atmosphere, coarse grinding of this alloy ingot was carried out using a jaw crasher and BURAUMMIRU in the argon ambient atmosphere, subsequently, the jet mill using nitrogen gas performed pulverizing, and R-Fe-B system magnet powder with a mean particle diameter of 5 micrometers was produced. The magnetic field of 15kOe(s) is impressed for this magnet powder, and it is 1 ton/cm2 to the magnetic field impression direction and a perpendicular direction. It fabricated by putting a pressure. Sintering was performed for this Plastic solid at 1060 degrees C in the argon ambient atmosphere for 90 minutes, aging heat treatment was performed to the pan by 540 ** after that, and it considered as the permanent magnet. The 30mmx20mmx10mm test piece was cut down from the obtained permanent magnet. After giving a metal plating layer

with the low degree of hardness described in this test piece among a table 1 at examples 1-5, respectively, the diameter threw the shot which is 15mm at the test piece surface, gave the mechanical shock, and covered the metal plating layer with the described various degrees of hardness high subsequently to a table 1. It is each covered test piece in the chamber of 60 degrees C and 95% relative humidity 600 Appearance was observed and corrosion resistance was evaluated, after carrying out time amount maintenance. A result is shown in a table 1.

[0016] It carried out like the example 1 - the example 5 except having not given a mechanical shock to the test piece cut down example of example of comparison 1- comparison 5. The result of these *****-proof is also described in a table 1.

[0017]

[A table 1]

	No.	硬度の低い金属めっき層		硬度の高い金属めっき層		&DEA(C ~ L) &D
		金属めっきの種類	膜厚 (µm)	金属めっきの種類	膜 厚 (µm)	試験後の外観
	ı	無電解Cu めっき	7	光沢Ni めっき	3	全く変化なし
実	2	無電解Cu めっき	7	無電解Ni - Pめっき	3	全く変化なし
施	3	無光沢Ni めっき	14	光沢Ni めっき	6	全く変化なし
例	4	無光沢Ni めっき	14	無電解Ni - Pめっき	6	全く変化なし
	5	無光沢Ni めっき	14	Ni ーFe 合金めっき	6	全く変化なし
	1	無電解 Cu めっき	7	光沢Ni めっき	3	ピンホール少数
比	2	無電解Cuめっき	. 7	無電解Ni - Pめっき	3	ピンホール少数
較	3	無光沢Ni めっき	14	光沢Ni めっき	6	ピンホール少数
例	4	無光沢Ni めっき	14	無電解Ni - Pめっき	6	ピンホール少数
	5	無光沢Ni めっき	14	Ni - Fe 合金めっき	6	ピンホール少数・

[0018] It carried out like the example 1 - the example 5 except having given the metal plating layer with a low degree of hardness to the test piece surface, having thrown the shot at the test piece surface and giving a mechanical shock, after cutting down six to example 10 test piece. The result of these ******-proof is shown in a table 2. [0019]

[A table 2]

	No.	硬度の低い金属めっき層		硬度の高い金属めっき層		E-PR-4ts on (a) les
	100.	金属めっきの種類	膜 厚 (µm)	金属めっきの種類	膜 厚 (µm)	試験後の外観
	6	無電解Cu めっき	7	光沢Ni めっき	3	全く変化なし
夷	7	無電解Cu めっき	7	無電解Ni - Pめっき	3	全く変化なし
施	8	無光沢 Ni めっき	14	光沢Ni めっき	6	全く変化なし
例	9	無光沢 Ni めっき	14	無電解Ni - Pめっき	6	全く変化なし
	10	無光沢Ni めっき	1 4	Ni - Fe 合金めっき	6	全く変化なし

[0020]

[Effect of the Invention] By this invention, the R-Fe-B system permanent magnet of high corrosion resistance can be obtained.

[Translation done.]

* NOTICES *

Japan Patent Office is not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.*** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] A high corrosion resistance permanent magnet characterized by carrying out the laminating of the metal plating layer with a high degree of hardness to a metal plating layer with a low degree of hardness on it on the surface of a R-Fe-B system permanent magnet (rare earth elements containing Y are kinds at least for R).

[Claim 2] A high corrosion resistance permanent magnet according to claim 1 whose metal plating layer with a low degree of hardness is a mat nickel-plating layer and whose metal plating layer with a high degree of hardness is a gloss nickel-plating layer.

[Claim 3] A high corrosion resistance permanent magnet according to claim 1 or 2 whose ratios of thickness of a metal plating layer and thickness of a metal plating layer with a high degree of hardness with a low degree of hardness are 6:4-8:2.

[Claim 4] A manufacture method of a high corrosion resistance permanent magnet characterized by giving a mechanical impact to this permanent magnet, crushing a pinhole of a metal plating layer with a low degree of hardness, and subsequently to a it top covering a metal plating layer with a high degree of hardness after covering a metal plating layer with a low degree of hardness on the surface of a R-Fe-B system permanent magnet (rare earth elements containing Y are kinds at least for R).

[Claim 5] A manufacture method of a high corrosion resistance permanent magnet characterized by covering a metal plating layer with a low degree of hardness on the surface of a R-Fe-B system permanent magnet (rare earth elements containing Y are kinds at least for R), crushing a pinhole of a metal plating layer with a low degree of hardness on it, giving a mechanical impact to this permanent magnet, and subsequently to a it top covering a metal plating layer with a high degree of hardness on it.

[Claim 6] A manufacture method of a high corrosion resistance permanent magnet according to claim 4 or 5 that a metal plating layer with a low degree of hardness is a mat nickel-plating layer, and a metal plating layer with a high degree of hardness is a gloss nickel-plating layer.

[Claim 7] A manufacture method of a high corrosion resistance permanent magnet according to claim 4 to 6 that ratios of thickness of a metal plating layer and thickness of a metal plating layer with a high degree of hardness with a low degree of hardness are 6:4-8:2.

[Translation done.]